

COAL RANK EFFECTS IN THE SRC II PROCESS

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INTRODUCTION

During the last two years the Merriam Laboratory has run a series of subbituminous coal samples in the SRC II operating mode^{1,2,3,4}. Recently, two samples of lignite have been run as well. These experiments have developed enough information to define a number of operating requirements for the low rank coals, and to give some indication of the kinds of differences inherent in these potential feedstocks. This paper gives results for coals representing bituminous, subbituminous and lignite ranks.

The SRC II process was developed with bituminous coals from the eastern United States. These coals were generally selected to contain sufficient iron pyrite to provide effective "self catalysis" and allow operation without use of additional catalyst. It is now well established that pyrite is rapidly converted to pyrrhotite and this is the catalyst which is used in this reaction. As an approximation, the response for a given coal appears to be proportional to the pyrite content of the coal. It also appears that coals with different organic phase compositions exhibit different levels of reactivity at similar iron or pyrite contents.

When western subbituminous coals or lignites are inspected, it is found that the pyrite content is usually low. On this basis, the prospect for adequate self catalysis is not favorable. These coals also tend to contain high bed moistures and high oxygen contents. Some oxygen is present in the form of carboxylate functions which are reacted with various mineral materials such as calcium, magnesium, sodium, or potassium. These materials can be removed by ion exchange procedures or by leaching with acid. Sometimes iron is present in leachable form, but most of the iron is usually present in pyrite form and the concentration is normally low.

When a subbituminous coal with ion exchangeable calcium is dissolved in single pass operations such as SRC I or by donor solvent procedures, interaction between calcium and the relatively high concentration of carbonate resulting from decomposition of the carboxylate functions causes calcium carbonate deposits to form in the reactor. These have proved troublesome according to reports from the Wilsonville and EDS Pilot Plants. We have also observed operating problems with Belle Ayr coal in the SRC I mode which appear to be due to carbonate precipitation. As a rule, the single pass procedure also fails to dissolve a significant fraction of the organic material fed with the coal. Insoluble organic matter yields of ten to twenty percent are often reported. Thus, the salt-like structures which are present appear to decompose at a slow rate compared to the rates observed with solvent swellable bituminous coals.

In our SRC II tests we have used addition of pyrite to correct for the low amount of iron present in the samples under consideration. With this kind of catalyst it has been found that conversions to distillate oil are high and that yields of insoluble organic matter are low. It has also been observed that carbonate precipitation is not a problem in this mode of operation. These tests have included pyrites from several sources and iron oxide plus elemental sulfur or carbon disulfide as catalyst. It appears that once an appropriate catalyst is used that the SRC II process will make distillate oil from subbituminous coals⁵ or lignites without difficulty.

This paper will present comparative results for runs made with Powhatan No. 3 Mine bituminous coal processed at 1800 psig (no added catalyst) and Blacksville No. 2

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Mine bituminous coal processed at 2250 psig with added pyrite. Two separate experiments with Belle Ayr subbituminous coal processed at 2250 psig will be reported. One run each with Texas Big Brown lignite and Indian Head lignite from North Dakota will be presented. Conventional operating conditions with well catalyzed bituminous coals require pressures of 1800 psig while exploratory work is usually done at 2250 psig to obtain a broader operating latitude.

SRC II EXPERIMENTAL PROCEDURE

The experimental results to be reported were obtained on a bench scale unit at Merriam using the equipment illustrated in Figure 1. This unit operates at a nominal feed rate of one kilogram of slurry per hour and in the SRC II mode processes a slurry containing 30% of coal (dry basis). The remainder of the slurry consists of reactor product (unfiltered coal solution), return oil, and catalyst as required. Generally, the ratio of unfiltered coal solution to return oil is adjusted to obtain a slurry with viscosity and solids which allow smooth pumping while the coal and catalyst content remains fixed. It is often necessary to warm the slurry to further adjust the viscosity of the feed slurry; therefore, the mix pot and the feed pot are traced with electrical heaters, as are all of the lines on the reactor system.

An experiment is started with a slurry of 30% coal and 70% coal-derived solvent. This is run through the reaction, and the product is mixed with coal in the ratio of 30% coal with the catalyst and unfiltered coal solution making up the balance. This is added to the feed pot and the procedure is continued until the solids level or the viscosity of the slurry require that the recipe be adjusted. At this point part of the unfiltered coal solution is displaced by return oil in the recipe. In this way it is possible to develop a steady state system in which the feed slurry solids will be maintained between 45% and 50% and which can be pumped at the required rate. When catalysts are added, they normally comprise one to two percent of the feed recipe. Of course, removal of gaseous products, water, and liquids too light for recycle causes the ash forming minerals and catalyst added to build up in concentration. The system is therefore interactive; the greater the yield of these volatile substances the greater the concentrating effect on the mineral phases. In the final steady state, the concentration of ash and catalyst is greater than the feed rate might suggest because of carry back of these materials with the unfiltered coal solution. The high temperature, high pressure separator is operated to obtain unfiltered coal solution which contains oils boiling over 250°C at atmospheric pressure. The secondary separators reclaim some oil in this boiling range which can be used as return oil and the remainder is made by distilling the unfiltered coal product not needed for feed formulation. In a plant the organic matter in the distillation residue would be used to make hydrogen for the process.

It can be seen that only coal is added to the system together with catalyst, where used, and that the oil in the initial step is soon diluted or reacted away. Only the coal being fed can be the source for oil in hand once the steady state has been sustained for an adequate period of time. The oil consists of any light product removed by the intermediate and low temperature separators which is not returned as part of the formulation plus oil distilled from the unfiltered coal solution and not reformulated. This distribution is subject to some adjustment depending on how the mechanics of the unit are managed. This depends on the solids level, the temperature of the feed pot, and the amount of oil returned to maintain the pumping characteristics of the feed slurry.

When steady state operation has been maintained for an adequate time, the output rate for each product category is measured. These categories are: gas, water, various separator streams, distilled oil from unfiltered coal solution, vacuum bottoms from this distillation and separation of the product into mineral and various soluble phases. From these data a yield is calculated referred to dry feed coal basis. All oils are analyzed by simulated distillation and a proportioned blend is made using the yields from each separator or distillation procedure (correcting for amounts returned to reaction). This blend of distillate oils then represents the

distillate oil made from the coal at the conditions of the experiment. Since there is no dilution with extraneous solvents, this blend should represent the input coal as operated on by the process and the catalyst in use.

The proportional blend is usually distilled to produce a naphtha, a middle oil, and a heavy oil sample. A retain sample of the proportional blend is also kept. This makes separate samples which can be analyzed in as much detail as time or equipment permits. This paper will concentrate on the preliminary description of these materials and a more detailed comparison will be developed later.

PREPARATION AND PROPERTIES OF FEED COALS

Our small unit requires coals which have been ground and sieved to eliminate particles which are larger than 150 mesh. This is required because of close tolerance on pump checks and letdown valves which will clog if the feed contains particles which are too large. All coal is therefore ground and then sieved before use. The final sieving is done in air, and, depending on conditions and the type of coal, a rather characteristic moisture content is observed. Typically, bituminous coal samples will equilibrate with about 1% of moisture while subbituminous coals will retain 7.5% to 10% moisture. Lignites which are managed in this way tend to retain about 18% to 20% moisture. These figures are less than the bed moisture or equilibrium moisture of the coals in question. The coals are partly dried during grinding and the sieving procedure usually has little further effect.

In our experiments coal samples are fed with these moisture contents and no attempt has been made to force complete drying. Drying is known to be detrimental for lignites and subbituminous coals because of the risk of oxidation of the material⁶. The usual bituminous coal will dry readily and does not retain troublesome amounts of water. Moisture is determined by toluene distillation rather than by oven drying. See ASTM D 95. This method gives higher moisture results than would be obtained by oven drying and the difference seems to be due to oxidation of the coal in the oven drying method. Slurry samples are taken during experiments and are analyzed for water by the toluene distillation method. A balance for water entering with the coal and water retained by the warm feed slurry is therefore possible. Loss of water from feed slurry is normally observed and its effect can be corrected in yield calculations.

Table I presents the compositional analysis of the coals chosen for this study. We are concerned with moisture content, the elemental analysis, the sulfur forms analysis, and the analysis of the mineral residue from ashing the sample. The free swelling index is useful for developing some intuition regarding the degree of polymerization of the bituminous coals, but the lower rank samples are nonagglomerating or have zero values. This probably indicates that the bonding is through mineral ions to a considerable extent. Typical small changes in moisture during sieving are shown.

Sulfur forms indicate the pyrite content of the coal. The total iron in the mineral residue will usually match the pyrite content closely or may run just a bit higher. The total amount of basic material in the ash is interesting because this relates to the tendency for the ash to retain sulfur when the coal is ignited. For many low rank coals much of the sulfur in the coal will be retained by the ash. Note that the Belle Ayr ash retains 20.78% of SO_3 and that the ash from the Texas Big Brown lignite retained 18.10% of SO_3 . Sulfur retention by the ash may vary in hydrogenation experiments if the sulfur removal during liquefaction is high. In this case, the sulfur remaining in the system may not produce an ash with a normal SO_3 content and corrections in the ash yield are necessary.

Elemental analysis results are sensitive to the method used for the determination of water, since most of these analyses have to be made on a wet or, at best, a partly dry sample. It is considered impractical to fully dry the low rank coals as partial oxidation may result. For these coals, results by the conventional ASTM oven drying

method are compared to results adjusted for the toluene distillation analysis. Since more water is generally found by the toluene distillation method, the hydrogen and oxygen content attributed to the organic phase will be changed by choice of this method. This change is also forced by the need to follow the water balance of the feed slurry by the distillation method.

In comparing P&M preferred results, the nitrogen value obtained by our laboratory will occasionally be found to be higher than the value reported by other laboratories. This higher result is usually justified by a better balance obtained in the nitrogen input with the coal and the nitrogen found in the product array. This difference is attributed to a more severe digestion procedure, particularly on the feed coal where a tendency for incomplete digestion is often observed. Products tend to digest more readily and anomalous results in which more nitrogen appears in the products than in the feed may be encountered. These differences are randomly encountered without regard for rank and probably reflect the experience of the analysts involved more than an inherent defect in the method. In these results, the Belle Ayr coal analysis is in disagreement.

YIELDS FROM SRC II REACTIONS

The results for each of the coal runs selected as examples are presented below to allow easy comparison. These are presented on a dry feed coal basis and catalyst conversion products are stated separately where catalysts are used.

PRODUCT YIELDS

Yields, Wt% based on MF Coal	DOE	Lignites		Subbituminous		Bituminous	
		Texas	No. Dakota	(Wyoming Belle Ayr)		Powhatan	Blacksville
Experiment Number	DOE	440RA	440RE	439RA**	439RD	409R	402R
Water		9.7	11.6	9.7	9.4	4.6	3.4
Carbon Monoxide		1.6	1.0	1.0	1.5	0.5	0.5
Carbon Dioxide		6.4	11.0	4.7	4.2	0.7	0.7
Hydrogen Sulfide*		2.2	1.9	1.5	1.7	3.1	2.7
Ammonia		0.8	0.6	0.7	0.6	0.4	0.5
Methane through Butanes		9.9	10.8	8.4	11.2	14.2	11.4
Naphtha (C ₅ through 193°C)		23.0	17.2	15.1	16.7	14.9	11.1
Middle Distillate (193-288°C)		23.1	19.3	19.2	24.1	16.5	13.6
Heavy Distillate (over 288°C)		5.8	4.4	15.6	11.3	8.5	12.8
Total Distillate		51.9	40.9	49.9	52.1	39.9	37.5
SRC (Pyridine sol., non-dist.)		12.7	16.3	21.1	17.0	25.2	31.7
Insoluble Organic Matter		0.9	1.8	3.7	3.1	5.4	5.4
Ash		11.0	9.8	6.6	6.8	10.2	12.5
Catalyst Conv. Products		2.1	2.1	2.1	2.0	none	2.1
Hydrogen Reacted		5.0	5.3	5.2	5.6	4.3	4.2
Reactor Pressure		2250	2250	2250	2250	1800	2250

* Hydrogen sulfide yield includes product from pyrite used as catalyst. All catalyzed trials used 5% pyrite based on coal fed. Powhatan and Blacksville are Pittsburgh seam (No. 8) coals.

** DOE 439RA run at 430°C for 1.55 hour retention time. All other runs are at 450°C for 1.0 hours nominal retention time, except DOE 409R run at 457°C for 1.0 hours.

The Powhatan No. 3 Mine coal could be processed at 1800 psig without the use of a catalyst since the coal contained enough pyrite to catalyze the reaction. The Blacksville Mine 2 coal is a less reactive coal run at conditions matching those used with the lignites and the DOE 439RD subbituminous trial.

A conventional reaction with a bituminous coal is run with a temperature profile in the reactor. This runs from 450°C at the bottom and reaches 460°C at the top. The volume average temperature is near 457°C. A baffled air furnace surrounds the reactor, therefore it is practical to impose this pattern on the reactor. This is done to match the profile observed in the Fort Lewis Pilot Plant's reactor. In explor-

tory work, the usual procedure is to maintain a flat profile at the specified temperature. Most of these runs were therefore done at a uniform 450°C, except for the DOE 439RA trial which used a uniform 430°C profile.

It appears that a temperature of about 450 to 455 centigrade is required for good operability with bituminous coals, and that somewhat lower temperatures are useful for the subbituminous coals and lignites. Experience is not extensive, but trials at 440°C and one hour nominal time produce good results. At temperatures lower than this, the reaction time must be extended to maintain the oil yield. Run DOE 439RA is an example of this kind of study. A small decrease in gas yield is observed when this change is made. A shift towards heavy oil and an increase in SRC is also observed.

As the oxygen content of the coal increased, a corresponding increase in the yield of carbon monoxide, carbon dioxide and water resulted. The lower rank coals appear to make a lighter average molecular weight product array (less bituminous material) and also to make a lower yield of pyridine insoluble material (insoluble organic matter, IOM). This trend increases as rank decreases from bituminous to subbituminous to lignite. Use of an adequate catalyst is implicit. When catalyst is dropped out of the feed for the Belle Ayr coal, the yield of IOM increases steadily during the time that the progressive dilution of the residual material in the feed takes place. Finally, the system becomes inoperable and the IOM at this point is about 14%⁵. Small amounts of iron seem effective for the low rank coals and the test of dropping out the catalyst feed did not produce a conclusive failure in the case of the Texas Big Brown lignite which contains 0.71% of iron. The North Dakota sample became inoperable when the pyrite addition was stopped. This sample contains 0.75% iron, therefore the difference is due to the difference in the organic phase. The Belle Ayr coal contains only 0.31% of iron and is clearly inoperable without a catalyst addition.

PRODUCT COMPOSITIONS

The main products derived from the SRC II process are the distillate oils. These are characterized by analysis of the three fractions distilled from the proportional blend representing all of the condensates and distillates made in the process. The elemental analysis of each fraction is presented in the following table for comparison.

It is clear that the compositions are much the same no matter what coal is under consideration. Small differences are observed which reflect the differing amounts of oxygen, sulfur and nitrogen in the organic phase of the coals and the tendency for a fraction of this material to be retained by the oil products. Oxygen is retained as an -OH functional group; compounds ranging from simple phenol through an assortment of methyl phenols and dimethyl phenols on to more complex substances. The mid range materials are the most abundant and oxygen tends to concentrate in the middle oil as phenolics. Where the oxygen input is large, the concentration of simple phenol is great enough for some oxygen to spill over into the naphtha fraction (the temperature was chosen while working with bituminous coals and is a little too high to cut out phenol cleanly).

It has been reported that the residual sulfur in SRC II oils is present in an array of thiophene derivatives⁷. This kind of structure has the thermal and chemical stability to survive at least in part at the conditions used. Sulfur elimination is much more complete than nitrogen elimination. Nitrogen is reported to remain in the oil as quinoline derivatives or other similar substances in which the nitrogen is in the ring⁸. About 3/4 of the total nitrogen is basic enough to titrate with perchloric acid in glacial acetic acid.

While distillation residues would normally be used for hydrogen manufacture feed-stock, it is recognized that they contain converted coal and that the inspection of

COMPOSITION OF PRODUCT OILS AND DISTILLATION RESIDUES

Analytical Result	Lignites		Subbituminous		Bituminous		
	Texas	No. Dakota	(Wyoming Belle Ayr)		Powhatan	Blacksville	
Experiment Number	OOE	440RA	440RE	439RA	439RD	409R	402R
Naphtha (IBP to 193°C)							
Carbon	83.30	81.11	84.75	84.98	86.11	86.01	
Hydrogen	13.20	12.25	13.20	13.44	12.63	13.26	
Sulfur	0.06	0.27	0.26	0.13	0.31	0.18	
Nitrogen	0.22	0.19	0.18	0.20	0.27	0.21	
Oxygen	3.22	6.18	1.61	1.25	0.68	0.34	
Middle Distillate (193-288)							
Carbon	84.70	83.77	84.15	84.97	85.40	85.08	
Hydrogen	9.55	9.10	9.26	9.62	9.05	9.41	
Sulfur	0.10	0.00	0.06	0.02	0.23	0.01	
Nitrogen	0.85	0.69	0.85	0.84	0.99	1.02	
Oxygen	4.80	6.44	5.68	4.55	4.33	4.46	
Heavy Distillate (over 288°C)							
Carbon	87.44	87.57	87.92	88.29	88.83	88.69	
Hydrogen	8.50	8.17	8.41	8.87	7.44	8.03	
Sulfur	0.15	0.14	0.06	0.05	0.78	0.29	
Nitrogen	1.19	1.05	1.07	1.13	1.25	1.22	
Oxygen	2.72	3.06	2.74	1.66	1.70	1.77	
Distillation Residue							
Carbon	45.40	54.96	63.85	59.85	65.40	60.24	
Hydrogen	2.93	3.10	4.03	3.52	3.90	3.74	
Sulfur*	5.80	5.75	4.55	5.24	3.38	4.38	
Nitrogen	0.95	0.95	1.19	1.06	1.33	1.23	
Ash**	52.59	43.54	30.18	35.45	26.76	39.61	

* Sulfur in distillation residue contains sulfur reacted with iron from catalyst.

** Ash contains catalyst residue. On oxidation to ash, substantial weight changes are produced which preclude any attempt to calculate oxygen content by difference.

the residue gives some insight into the chemistry of the process. For that reason a number of extractions are done to further characterize the material. The ash consists of the reduced mineral matter fed with the coal and derived from the pyrite where this catalyst is added. When analyzed, the total sulfur in the mineral phase contributes much of the value and obscures the composition of the organic material. The weight gain of the mineral phase on ignition precludes calculation of the oxygen content of the organic phase by difference. It is possible to extract sequentially with appropriate solvents and obtain the organic phase as a separated material for analysis, but this is not routinely done.

Distillation residues are routinely characterized by extraction with hexane, toluene and pyridine. These are run in Soxhlet extractors using ceramic thimbles at near the boiling point of each solvent. The residue is ground and sieved to -100 mesh before the extraction procedure. Usually the work is done with a single solvent applied to each of three samples. Results for the residues are expressed in weight percent soluble and also as the fraction of the total soluble in pyridine which is soluble in hexane or in toluene. IOM is defined as pyridine insoluble organic matter in our procedures.

It is also possible to calculate a distribution by subtraction of values obtained by extraction. The hexane soluble non-distillable fraction (which has been called maltene by some workers⁹ is obtained directly. By subtraction of the hexane solubility from the toluene solubility, the amount of hexane insoluble but toluene soluble material is calculated. Again, by subtraction of the toluene soluble term from the pyridine soluble term, the amount of toluene insoluble but pyridine soluble material can be calculated. The values correspond, somewhat loosely, to maltene, asphaltene, and pre-asphaltene. Results for these extraction procedures follow:

EXTRACTION RESULTS FOR DISTILLATION RESIDUES

Analytical Result		Lignites		Subbituminous		Bituminous	
		Texas	No. Dakota	(Wyoming Belle Ayr)		Powhatan	Blacksville
Experiment Number	DOE	440RA	440RE	439RA	439RD	409R	402R
<u>Solubility in</u>							
Hexane, wt %		26.6	24.5	36.3	28.7	23.9	30.9
Toluene, wt %		40.3	44.0	52.7	49.7	52.4	53.8
Pyridine, wt %		43.6	51.6	61.7	55.3	59.8	58.2
Pyridine Insoluble, %		56.4	48.4	38.3	44.7	40.2	41.8
<u>Fractions</u>							
Hexane/pyridine		0.610	0.469	0.588	0.519	0.400	0.530
Toluene/pyridine		0.925	0.835	0.854	0.898	0.876	0.926
<u>Yield of Fractions, wt %</u>							
Hexane Soluble		7.74	9.90	12.41	8.83	10.08	16.80
Toluene Soluble		4.00	7.72	5.61	6.44	12.00	12.52
Pyridine Soluble		0.95	3.48	3.08	1.73	3.12	2.35
Total SRC Solids		12.7	21.1	21.1	17.0	25.2	31.7
Pyridine Insoluble		0.9	1.8	3.7	3.1	5.4	5.4

The recycle procedure used in the SRC II process exposes the non-volatile products derived from coal to reaction conditions repeatedly. When catalysis is adequate to allow prompt quenching after thermal bond breaking, the heavy material is reduced in average molecular weight (or at least made more soluble). Thus, the solubility of the non-distillable material in hexane at reflux will be an appreciable fraction of the organic material which is soluble in the best available solvent (pyridine). Most of the organic material remaining is soluble in toluene at reflux. The concentration of pre-asphaltene is low in all cases. These results are significantly different from single pass results of the SRC I process. In that case, retention times and pressures are adjusted to make non-distillable material as the principal product and hexane solubilities are often not more than 20% or so of the SRC solids obtained. Toluene solubility is less in that case also.

DISCUSSION

These experiments have highlighted two fundamental analytical problems in the conduct of liquefaction runs with low rank coals. First, the determination of water by conventional oven drying methods leads to difficulties in matching water input and water content of slurries. When all determinations are based on toluene distillation, rational values are obtained, but a shift in hydrogen and oxygen content attributed to the organic phase of the coal is introduced. The difference between oven drying and the toluene distillation (actually xylene distillation is the recommended method) is well documented¹⁰. These distillation methods are reported to be the more accurate method and our experience is in agreement.

A less well known problem was first well defined when molybdenum was used as a catalyst with low rank coals. This can be effective at quite low concentrations and contributes little to the ash content of the system. Difficulty was observed in matching ash input and outputs, and even after long equilibration periods, the recovery of ash in products was low. Analysis of the ash for sulfur indicated that less SO₂ was present in the ash than would be found in the ash from the ASTM analysis. This seems reasonable when the evolution of sulfur as hydrogen sulfide during reaction is considered. Correction of the ash to a new weight basis with the sulfur correction brought input and output into balance. Pyrrhotite seems to ignite to ferric oxide without complications; therefore, the problem is less evident in those cases in which pyrite is used as a catalyst. The effect may crop up in some instances where a catalyst is dropped out and the reaction can be sustained down to low levels of residual catalyst, as in the case of the Texas lignite.

While data are not extensive, it appears that conversion is highest for lignites and a little lower for subbituminous coals. Most bituminous coals tend to give even lower conversions. Thus, IOM yields for the lignites tend to be below 2%, for the

subbituminous coals in the range from 2% to 4%, while the bituminous coals range 4% and up to perhaps 7%. The IOM yield is sensitive to catalysis, pressure, temperature and coal type, with yields in a well managed SRC II operation tending toward the lower limits for a given coal.

Oxygen and sulfur removal is reasonably effective for coals of all ranks in the SRC II process, but nitrogen removal is quite incomplete. The main driving force is thermal bond breaking and none of the disposable catalysts which have been tested have significantly improved the nitrogen removal situation. The product compositions which are presented are typical¹¹.

At the conditions used in exploratory work, yields of oil are high with the low rank coals. Conversely, the yield of bituminous residue is lower than normal and is actually lower than the amount needed for hydrogen manufacture.

Pyrites are the best disposable catalyst now identified and application to low rank coals at optimum conditions may become attractive. The effectiveness of pyrite is much more evident in recycle than in single pass procedures. This may be due to the time required for dispersion of the material or other factors not understood at this time. Addition of pyrite to bituminous coals may not appear effective in those cases where quite a bit of iron is already present. It also appears that the structure of the organic phase is an important factor which limits the rate of conversion to oil. Thus, the system becomes insensitive to catalysis unless some factor which influences cracking is also present. It does not appear that iron sulfide facilitates cracking of the heavy materials but rather functions to maintain a favorable concentration of hydrogen in the liquid phase to favor radical quenching reactions. If this is true, the low rank coals must yield smaller and relatively simpler intermediate reaction products because of the relative ease with which distillate is obtained.

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FIGURE 1
MERRIAM LABORATORY
BENCH SCALE COAL LIQUEFACTION UNIT

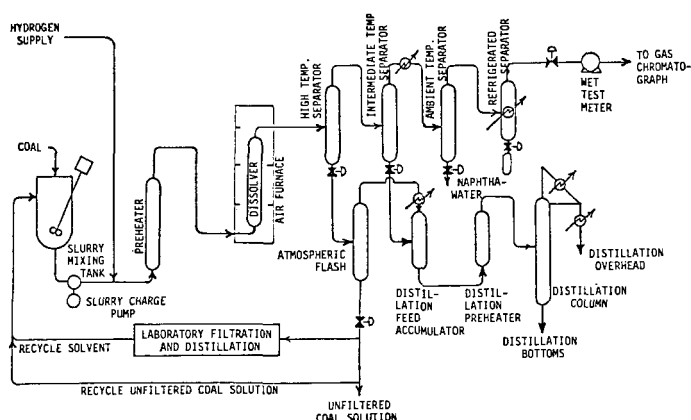


TABLE I
Coal Analyses

Coal Identification	Texas Utility	North American	Anax	North American	Consol
Company	Big Brown	Indian Head	Belle Ayr	Powhatan No. 3	Blacksville No. 2
Mine	Wilcox	Beula-Zap	Wyodak-Anderson	Pittsburgh	Pittsburgh
Seam			Wyoming	Pennsylvania	Pennsylvania
State	Texas	North Dakota			
Rank	Lignite	Lignite	Subbituminous	Bituminous	Bituminous
Proximate Analysis					
% Ash	11.44	10.15	7.16	10.85	11.94
% Volatile	47.19	45.24	41.78	42.29	38.69
% Fixed Carbon	41.37	44.61	51.06	46.86	49.37
% Moisture ASTM D 3173	17.98	13.88	6.71	3.72	2.32
% Moisture ASTM D 95	18.37 21.58	15.10 19.63	6.06 8.50	1.30 1.90	0.92 1.54
Heating Value, BTU/lb	10,977	10,067	11,951	13,171	13,331
Free Swelling Index	0	0	0	6	8
Sulfur Forms					
% Pyrite Sulfur	0.14	0.47	0.16	2.03	1.65
% Sulfate Sulfur	0.03	0.19	0.05	0.08	0.05
% Organic Sulfur	0.93	0.40	0.43	2.54	1.39
% Total Sulfur	1.10	1.06	0.64	4.65	3.09
Ultimate Analysis, dry basis					
% Carbon	63.85	61.80	69.80	72.03	71.10
% Hydrogen	4.93	4.13	4.84	5.11	5.22
% Nitrogen	1.19	0.89	0.90	1.17*	1.55
% Chlorine	0.07	0.03	0.15	0.09	0.07
% Sulfur	1.10	1.06	0.64	4.65	3.08
% Ash	11.44	10.15	7.16	10.85	11.94
% Oxygen	17.42	21.94	16.51	5.97	7.04
Mineral Analysis, wt %					
Ignited Basis					
% Silica, SiO ₂	34.85	21.49	27.50	41.62	42.60
% Alumina, Al ₂ O ₃	12.78	8.75	16.26	20.19	20.02
% Titania, TiO ₂	1.18	0.37	1.26	0.89	0.91
% Ferric Oxide, Fe ₂ O ₃	8.90	10.78	6.26	28.24	20.86
% Lime, CaO	19.16	16.05	20.24	2.65	6.06
% Magnesia, MgO	3.32	5.09	3.40	0.68	0.94
% Potassium Oxide, K ₂ O	0.38	0.80	0.74	1.59	1.49
% Sodium Oxide, Na ₂ O	0.66	9.78	1.34	0.87	1.20
% Sulfur Trioxide, SO ₃	18.10	25.52	20.87	3.03	5.33
% Phos. Pentoxide, P ₂ O ₅	0.04	0.19	1.25	0.11	0.48
% Strontium Peroxide	0.33	0.60	0.41	0.04	0.00
% Barium Oxide	0.04	0.56	0.45	0.04	0.08
% Manganese Oxide	0.26	0.02	0.02	0.05	0.04
% Undetermined	--	--	--	--	--
% Iron in Coal	0.71	0.76	0.31	2.14	1.74
% Equilibrium Moisture	26.28	32.39	23.06	2.66	

* Nitrogen value in disagreement.

Toluene distillation result only available on sieved samples.

Commercial analysis results are adjusted for difference in water by D 3173 & D 95.

Sieved samples may contain slightly different ash values and yields are adjusted to the analysis for specific lots of coal fed.